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IS 380 (1978): French chalk, technical [CHD 1: Inorganic Chemicals]



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“Knowledge is such a treasure which cannot be stolen”



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*Indian Standard*  
SPECIFICATION FOR  
FRENCH CHALK, TECHNICAL  
( *Second Revision* )

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**INDIAN STANDARDS INSTITUTION**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

*Indian Standard*  
**SPECIFICATION FOR**  
**FRENCH CHALK, TECHNICAL**  
*( Second Revision )*

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*Indian Standard*  
SPECIFICATION FOR  
FRENCH CHALK, TECHNICAL  
( *Second Revision* )

**0. FOREWORD**

**0.1** This Indian Standard ( Second Revision ) was adopted by the Indian Standards Institution on 15 September 1978, after the draft finalized by the Inorganic Chemicals ( Miscellaneous ) Sectional Committee had been approved by the Chemical Division Council.

**0.2** French chalk is largely used in the preservation of rubber goods, in pyrotechnic compositions, as a filler for insecticides, in the manufacture of paper and as a sizing material in textile.

**0.2.1** French chalk is also used by paint industry and in the production of cosmetics and high frequency ceramics but the material for these end uses is not covered in this standard.

**0.3** This standard was first issued in 1952 and revised in 1967. In the first revision the requirements of iron, relative density, and particle size, grit and whiteness were added. In the present revision changes have been made in the methods of determination of relative density, chlorides, iron and manganese.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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**1. SCOPE**

**1.1** This standard prescribes the requirements and the methods of sampling and test for French chalk, technical, used for various purposes.

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\*Rules for rounding off numerical values ( *revised* ).

1.1.1 This standard does not cover the material required for paint industry, cosmetics and high frequency ceramics.

## 2. REQUIREMENTS

**2.1 Description** — French chalk, technical, shall be obtained from the natural mineral (steatite, talc or soapstone). It shall be in the form of dry, fine, white powder, free from extraneous impurities, except small quantities of substances naturally occurring in the mineral. It shall have a smooth and soapy feel.

**2.2 Free Moisture and Volatile Matter** — The free moisture and volatile matter in the material, when determined according to the method prescribed in Appendix A, shall not exceed 0.5 percent by mass.

**2.3 Particle Size** — The material shall have no residue when passed through 75-micron IS Sieve (see IS: 460-1962\*) or as mutually agreed to between the purchaser and the supplier.

**2.4** The material, dried according to the methods prescribed in Appendix A, shall also comply with the requirements in Table 1, when tested according to the methods prescribed in Appendix B. Reference to the relevant clauses of Appendix B is given in col 4 of the table.

**TABLE 1 REQUIREMENTS FOR FRENCH CHALK, TECHNICAL**

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL No. IN APPENDIX B
(1)	(2)	(3)	(4)
i)	Loss on ignition, percent by mass, <i>Max</i>	4	B-3
ii)	Matter insoluble in hydrochloric acid, percent by mass, <i>Min</i>	95	B-4
iii)	Relative density at 27/27°C	2.7 to 2.9	B-5
iv)	Chlorides (as NaCl), percent by mass, <i>Max</i>	0.5	B-6
v)	pH (of 10 percent solution), <i>Max</i>	8.5	B-7

**2.5 Additional Requirements** — The material, required for use in textile, paper and pyrotechnic industries, shall, in addition to the requirements prescribed in Table 1, also comply with the requirements prescribed in Table 2 when tested according to the methods prescribed in Appendix B. Reference to the relevant clauses of Appendix B is given in col 4 of the table.

\*Specification for test sieves (*revised*).



**TABLE 2 ADDITIONAL REQUIREMENTS FOR FRENCH CHALK,  
TECHNICAL, FOR USE IN TEXTILE, PAPER AND  
PYROTECHNIC INDUSTRIES**

Sl No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO Cl. No. IN APPENDIX B
(1)	(2)	(3)	(4)
i)	Grit, percent by mass, <i>Max</i>	0.02	B-8
ii)	Iron ( as $\text{Fe}_2\text{O}_3$ ), percent by mass, <i>Max</i>	0.3	B-9
iii)	*Whiteness, reflectance to blue light of wavelength 5 040 Å, percent, <i>Min</i>	80	B-10

\*This requirement shall be tested only when required by the purchaser.

**2.6 Copper, Manganese and Their Compounds** — The material, required for use in the preservation of rubber goods, shall contain not more than 0.05 percent by mass of either copper or manganese or their compounds calculated in terms of respective metals, when tested according to the method prescribed in Appendix C.

### 3. PACKING AND MARKING

**3.1 Packing** — The material shall be packed in paper bags or hessian bags with a suitable liner. For special requirements, the packing may be as agreed to between the purchaser and the supplier. The packages shall be securely closed.

**3.2 Marking** — The packages shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Gross and net mass;
- d) Date of packing; and
- e) Batch number.

**3.2.1** The packages may also be marked with the ISI Certification Mark.

**NOTE** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

#### 4. SAMPLING

4.1 The method of drawing representative samples of the material, number of tests to be carried out and the criteria for conformity of the material in a lot to the requirements of the specification shall be as prescribed in Appendix D.

### APPENDIX A

( Clause 2.2 )

#### DETERMINATION OF FREE MOISTURE AND VOLATILE MATTER

##### A-1. PROCEDURE

A-1.1 Introduce about 5 g of the material in a tared weighing bottle provided with a glass stopper and weigh. Remove the stopper and heat for 3 hours at 105 to 110°C. Cool in a desiccator, replace the stopper and weigh. Repeat the process for one hour till constant mass is obtained.

##### A-2. CALCULATION

Free moisture and volatile matter, 
$$\text{percent by mass} = \frac{M_1}{M_2} \times 100$$

where

$M_1$  = loss in mass in g of the material after heating, and

$M_2$  = mass in g of the material taken for the test.

### APPENDIX B

( Clauses 2.4 and 2.5 )

#### METHODS OF TEST FOR FRENCH CHALK, TECHNICAL

##### B-1. QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals and distilled water ( see IS : 1070-1977\* ) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

##### B-2. PREPARED SAMPLE

B-2.1 Take about 50 g of the material in a china dish and dry to constant mass at 105 to 110°C. Cool and keep this *prepared sample* in a desiccator for subsequent tests.

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\*Specification for water for general laboratory use ( *second revision* ).

### B-3. DETERMINATION OF LOSS ON IGNITION

**B-3.1 Procedure** — Weigh accurately about 5 g of the *prepared sample* (see B-2) in a silica or porcelain crucible of 25 ml capacity and heat in a muffle furnace at 850 to 900°C. Cool to about 100°C and then finally transfer the crucible with its contents to a desiccator till it comes down to room temperature and weigh. Repeat the process until constant mass is obtained.

#### B-3.2 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

$M_1$  = loss in mass in g of the material after ignition, and

$M_2$  = mass in g of the *prepared sample* taken for the test.

### B-4. DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID

#### B-4.1 Reagents

**B-4.1.1 Rectified Spirit** — See IS : 323-1959\*.

**B-4.1.2 Dilute Hydrochloric Acid** — Approximately 5 N.

**B-4.2 Procedure** — Weigh accurately about 0.5 g of the *prepared sample* (see B-2) and transfer to a 200-ml beaker. Add 5 ml of rectified spirit so that the material is thoroughly wetted, and then add 100 ml of dilute hydrochloric acid. Boil for 10 minutes. Filter off the insoluble matter through a tared sintered glass crucible (G No. 4). Wash the residue with water till it is free from chlorides and dry at  $140 \pm 5^\circ\text{C}$  for one hour. Cool in a desiccator and weigh till constant mass is obtained.

#### B-4.3 Calculation

$$\text{Matter insoluble in hydrochloric acid, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

$M_1$  = mass in g of the residue obtained, and

$M_2$  = mass in g of the *prepared sample* taken for the test.

### B-5. DETERMINATION OF RELATIVE DENSITY

#### B-5.1 Apparatus

**B-5.1.1 Relative Density Bottle** — 50 ml capacity, having a ground-in capillary stopper with square end.

\*Specification for rectified spirit (*revised*).

**B-5.1.2 Vacuum Pump** — with a receiver, capable of giving a vacuum of 100 mm.

**B-5.2 Procedure** — Weigh the empty, dry relative density bottle, introduce about 10 g of the material and weigh again. Nearly fill the relative density bottle with water and do not insert the stopper. Place it on a small tared evaporating dish and keep the assembly in the exhausted receiver of the vacuum pump overnight (at least 16 hours). Fill up the relative density bottle carefully with kerosine (see IS: 1459-1974\*) and insert the stopper. It is possible that a very small portion of the sample will run out on the sides of the stopper on inserting it. Wash down the particles thus forced out into the tared dish. Wipe off the excess of kerosine from the relative density bottle, bring it to 27°C and weigh.

**B-5.2.1** Evaporate the contents of the tared dish on a boiling water-bath under cover, dry to constant mass at 105 to 110°C.

**B-5.2.2** Empty the specific gravity bottle, fill it with kerosine and weigh after bringing it to 27°C.

### B-5.3 Calculation

$$\text{Relative density at } 27/27^{\circ}\text{C} = \frac{M_2 - M_1 - M_4}{(M_5 - M_1) - (M_3 - M_2) - M_4} \times d$$

where

$M_2$  = mass in g of relative density bottle and sample,

$M_1$  = mass in g of empty relative density bottle,

$M_4$  = mass in g of residue in tared dish,

$M_5$  = mass in g of relative density bottle and water,

$M_3$  = mass in g of relative density bottle with sample and water,  
and

$d$  = relative density of kerosine.

## B-6. DETERMINATION OF CHLORIDES

### B-6.1 Apparatus

**B-6.1.1 Nessler Cylinders** — 50 ml capacity (see IS: 4161-1967†).

### B-6.2 Reagents

**B-6.2.1 Dilute Nitric Acid** — 5 N approximately.

**B-6.2.2 Silver Nitrate Solution** — 10 percent.

**B-6.2.3 Standard Hydrochloric Acid** — 0.01 N.

\*Specification for kerosine (second revision).

†Specification for Nessler cylinders.

**B-6.3 Procedure** — Weigh accurately about 1 g of the *prepared sample* (see B-2) and shake with about 10 ml of water. Filter through a folded filter paper and wash thoroughly with water, collecting the filtrate in a Nessler cylinder. Add 10 ml of dilute nitric acid, dilute to 50 ml with water and add 1 ml of silver nitrate solution. Stir immediately with a glass rod and allow to stand for 5 minutes.

**B-6.3.1** Place 1 ml of standard hydrochloric and 10 ml of dilute nitric acid in another Nessler cylinder. Dilute to 50 ml with water and add 1 ml of silver nitrate solution. Stir immediately with glass rod, and allow to stand for 5 minutes.

**B-6.3.2** The material shall be considered to have not exceeded the limit for chloride prescribed in Table 1 if the opalescence produced in the test solution is not greater than that produced in the control test.

## B-7. DETERMINATION OF pH

**B-7.1 Procedure** — Shake 10.0 g of the *prepared sample* (see B-2) with 100 ml of freshly boiled and cooled water for one minute and determine the pH by means of a pH meter using glass electrode.

## B-8. DETERMINATION OF GRIT

**B-8.1 Procedure** — Measure a depth of 50 mm from the bottom of a 500-ml beaker of about 8 cm diameter and make a mark on the beaker to indicate this height. Weigh accurately about 20 g of the material into the beaker. Break up any lump of the material with a rubber policeman but no hard object should be used for this purpose which may crush the particles of grit. Mix the material thoroughly with water and fill up to the mark. Allow to settle for exactly one minute and pour off the milky water. Repeat the process until the supernatant water can be poured off practically clean at the end of one minute. Place the beaker on the water-bath until perfectly dry. Brush out the settled grit into a tared watch glass with camel hair brush and weigh. Examine the material under microscope after weighing to confirm the presence of grit.

**B-8.2 Calculation** — Express the mass of grit as the percentage on the mass of the material taken for the test.

## B-9. DETERMINATION OF IRON

### B-9.1 Apparatus

**B-9.1.1 Nessler Cylinders** — 50 ml capacity (see IS : 4161-1967\*).

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\*Specification for Nessler cylinders.

## B-9.2 Reagents

**B-9.2.1 Concentrated Hydrochloric Acid** — See IS : 265-1976\*.

**B-9.2.2 Ammonium Persulphate**

**B-9.2.3 Butanolic Potassium Thiocyanate Solution** — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

**B-9.2.4 Standard Iron Solution** — Dissolve 0.4911 g of ferrous ammonium sulphate [  $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  ] in 10 ml of dilute sulphuric acid ( 10 percent by volume ) and dilute with water to 1 000 ml. One millilitre of this solution is equivalent to 0.1 mg of iron ( as  $\text{Fe}_2\text{O}_3$  ).

**B-9.3 Procedure** — Digest 1.0 g of the *prepared sample* ( see B-2 ) with 5 ml of water and 15 ml of concentrated hydrochloric acid for one hour, on a hot-plate without boiling. Cover the beaker in which digestion is done, with a watch glass containing some water. After the digestion is over, wash the under-side of the watch glass with water into the beaker. Filter and wash the residue six times with hot water adding the washings to the filtrate. Make up the volume of the filtrate to 250 ml with water. Pipette out 25 ml of this solution in a Nessler cylinder, add 50 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution and make up the solution to 50-ml mark with water. Carry out a control test in another Nessler cylinder with 3 ml of standard iron solution and the same quantities of other reagents in exactly the same way as above.

**B-9.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour obtained in the test with the material is not greater than that in the control test.

## B-10. DETERMINATION OF WHITENESS

**B-10.0 Principle** — The colour of the sample is compared with that of Lovibond magnesium oxide block with the help of a suitable apparatus.

**B-10.1 Procedure** — By means of a suitable arrangement, light falling on a Lovibond magnesium oxide block shall be reflected on a selenium cell connected to a galvanometer. The galvanometric deflection is proportional to light reflected. For comparison the reading obtained by magnesium oxide block is taken as 100.

**B-10.1.1** Substitute the magnesium oxide block with a block prepared from a sample of the material. Measure the reflectance and report as whiteness ( or reflectance ) compared to that of magnesium oxide as 100.

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\*Specification for hydrochloric acid ( *second revision* ).

## APPENDIX C

( Clause 2.6 )

## DETERMINATION OF COPPER AND MANGANESE

## C-1. PREPARATION OF THE SOLUTION

## C-1.1 Reagents

C-1.1.1 *Anhydrous Sodium Carbonate* — See IS : 296-1974\*.C-1.1.2 *Concentrated Sulphuric Acid* — See IS : 266-1977†.C-1.1.3 *Concentrated Nitric Acid* — See IS : 264-1976‡.

**C-1.2 Procedure** — Grind the material fine enough to pass through a 106-micron IS Sieve. Weigh accurately 1 g of the finely ground material and mix it with 4 g of anhydrous sodium carbonate and fuse in a crucible until the melt is clear. Pour the hot fused mass in a dish and rotate so as to form a thin film, and then put the crucible in the dish with the fused mass. Add about 10 ml of water and heat on the water-bath until the product is disintegrated. Remove the crucible and wash with water. Add 5 ml of concentrated hydrochloric acid in the crucible and digest hot, until the iron stain in it dissolves out completely. Add this acid to the contents in the dish. Evaporate to near dryness. Add a mixture of 6 ml of concentrated sulphuric acid and 1 ml of concentrated nitric acid. Evaporate to fumes of sulphur trioxide ( $\text{SO}_3$ ). Cool and dilute to exactly 250 ml in a volumetric flask. Use this *prepared solution* for the tests under **C-2** and **C-3**.

## C-2. DETERMINATION OF COPPER

## C-2.1 Apparatus

C-2.1.1 *Nessler Cylinders* — 100 ml capacity ( see IS : 4161-1967§ ).

## C-2.2 Reagents

C-2.2.1 *Ammonium Hydroxide* — approximately 1 : 4 ( v/v ).

**C-2.2.2 Sodium Diethyldithiocarbamate Solution** — Dissolve 1 g of sodium diethyldithiocarbamate in 1 000 ml of water. Keep in an amber coloured glass bottle and store in a dark place. This solution should not be used after two weeks.

\*Specification for anhydrous sodium carbonate ( *second revision* ).†Specification for sulphuric acid ( *second revision* ).‡Specification for nitric acid ( *second revision* ).

§Specification for Nessler cylinders.



**C-2.2.3 Gum Arabic Solution** — one percent (m/v).

**C-2.2.4 Standard Copper Solution** — Dissolve 0.3928 g of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), equivalent to 0.1000 g of copper in water. Make up the volume to 1000 ml with water. Pipette 100 ml of this solution into a 1000-ml volumetric flask and dilute up to the mark with water. One millilitre of this solution contains 0.01 mg of copper (as Cu). This dilute solution should be freshly prepared before use.

**C-2.3 Procedure** — Pipette 25 ml of the *prepared solution* into a small conical flask or beaker. Drop into it a small piece of litmus paper and make the solution just alkaline with ammonium hydroxide. Add 2.5 ml of ammonium hydroxide in excess and heat to boiling. Allow to stand on a water-bath for one hour to ensure complete precipitation of aluminium hydroxide and then filter through Whatman filter paper No. 1 or equivalent into a Nessler cylinder, washing the filter paper with two or three small portions of hot water. To the solution in the Nessler cylinder add 1 ml of gum arabic solution, 10 ml of ammonium hydroxide and 10 ml of sodium diethyldithiocarbamate solution. Dilute to 100-ml mark and mix well. To the other Nessler cylinder containing a similar aliquot blank solution carried through the entire analysis in the same manner as the *prepared solution*, add equal amounts of the same reagents, dilute to about 90 ml and mix. Titrate this solution with standard copper sulphate solution from a 10-ml burette until its colour matches that of the prepared solution when diluted to the same volume. Mix well after each addition of standard copper sulphate solution.

**C-2.3.1** If the colour produced with the *prepared solution* is too deep for comparison, a smaller aliquot portion of the *prepared solution* from the acid digestion shall be used.

**C-2.3.2** In case the *prepared solution* is turbid, due to the presence of some interfering substances like iron, lead, bismuth or zinc, the following modified procedure is recommended.

**C-2.3.2.1** If iron is present, precipitate the iron by adding a slight excess of ammonium hydroxide, filter and wash. Dissolve the ferric hydroxide precipitate in dilute nitric acid, precipitate with ammonium hydroxide, filter and wash. Combine the two filtrates, and dilute to a known volume. Follow rest of the procedure as under **C-2.3**.

**C-2.3.2.2** If lead or bismuth is present, add a few drops of ferric chloride solution, boil and precipitate twice with ammonium hydroxide as under **C-2.3.2.1**. Follow rest of the procedure as under **C-2.3**.

**C-2.3.2.3** If zinc is present, add sufficient ammonium hydroxide solution to prevent any turbidity developing after the addition of the reagent, and then follow the rest of the procedure as under **C-2.3**.

**C-2.4 Calculation**

$$\text{Copper, percent by mass} = 0.01 \times \frac{V}{M}$$

where

$V$  = volume in ml of standard copper sulphate solution used for the titration, and

$M$  = mass in g of the material taken under **C-1.2**.

**C-3. DETERMINATION OF MANGANESE****C-3.1 Apparatus**

**C-3.1.1 Nessler Cylinders** — 100 ml capacity (see IS : 4161-1967\*).

**C-3.2 Reagents**

**C-3.2.1 Phosphoric Acid** — 85 percent by mass.

**C-3.2.2 Potassium Periodate**

**C-3.2.3 Standard Potassium Permanganate Solution** — Prepare a dilute solution of potassium permanganate by diluting to 50 ml in a volumetric flask a quantity of recently standardized solution in accordance with the following formula:

$$V = \frac{0.4551}{N}$$

where

$V$  = volume in ml of standard potassium permanganate solution to be made up to a volume of 50 ml, and

$N$  = normality of the standard potassium permanganate solution.

NOTE — Do not keep the solution for more than one week. One millimetre of this solution is equivalent to 0.1 mg of manganese (as Mn).

**C-3.3 Procedure** — Transfer 100 ml of the prepared solution to a 250-ml beaker, evaporate to 75 ml and add 10 ml of phosphoric acid. Sprinkle 0.5 g of potassium periodate into the solution and bring it to boil. Go on adding 0.1 g more potassium periodate every 3 to 4 minutes until the permanganate does not seem to deepen any more and then add 0.1 g more. Place the beaker over a boiling water bath for 15 minutes. Cool and transfer to a Nessler cylinder and dilute to mark with water.

**C-3.3.1** In the second Nessler cylinder, place 2 ml of standard potassium permanganate solution and 10 ml of phosphoric acid and dilute to mark with water. Mix the contents of the tubes well before matching.

**C-3.3.2** The material shall be deemed to have passed the test if the colour produced by the test sample is not deeper than that produced by the standard solution.

\*Specification for Nessler cylinders.

## APPENDIX D

( Clause 4.1 )

### SAMPLING OF FRENCH CHALK, TECHNICAL

#### D-1. GENERAL REQUIREMENTS OF SAMPLING

**D-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**D-1.1** Samples shall not be taken in an exposed place.

**D-1.2** Sampling instrument shall be clean and dry when used.

**D-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination and harmful contact and exposure.

**D-1.4** The samples shall be placed in clean, dry, air-tight containers made of glass or other suitable material.

**D-1.5** The sample containers shall be of such a size that they are almost completely filled by the sample.

**D-1.6** Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sample, the date of sampling and other identification particulars.

**D-1.7** The samples shall be stored in such a manner that during storage the properties of the material do not get affected.

**D-1.8** Sampling shall be done by a person or persons mutually agreed to between the purchaser and the supplier.

#### D-2. SCALE OF SAMPLING FOR MATERIAL PACKED IN CONTAINERS

**D-2.1 Lot** — In a single consignment all containers of the material coming from the same source or supplier, shall constitute a lot.

**D-2.2 Sub-lot** — For the purpose of sampling, all the material in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal weight in accordance with col 1 and 2 of Table 3.

**D-2.3** The number of containers to be sampled from each sub-lot is given in col 3 of Table 3. These containers shall be selected at random from the sub-lot. From each of these containers a small but equal increment

of material shall be taken. In order to ensure the randomness of selection, necessary guidance may be obtained from IS : 4905-1968\*.

**TABLE 3 SCALE OF SAMPLING FOR CONTAINERS**( *Clauses D-2.2 and D-2.3* )

NO. OF CONTAINERS IN THE LOT	NO. OF SUB-LOTS	NO. OF CONTAINERS TO BE SAMPLED FROM EACH SUB-LOT
(1)	(2)	(3)
Up to 10	1	All
11 „ 200	2	5
201 „ 500	3	10
501 „ 2 001	4	15
2 001 and above	5	20

**D-2.4** All the increments from the sample containers in the same sub-lot shall be pooled together to constitute a gross sample so that there are as many gross samples as the sub-lots into which a lot has been divided.

### D-3. SAMPLING FROM BULK

**D-3.1 Lot** — The part or whole of the material in the bulk coming from the same source or supplier shall constitute a lot.

**D-3.2 Sub-lot** — For the purpose of sampling, the material in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal weight in accordance with Table 4 by suitably marking the lines of demarcation on the surface of the bulk.

**TABLE 4 SCALE OF SAMPLING FOR BULK AND WAGONS**( *Clauses D-3.2 and D-3.3* )

WEIGHT IN THE BULK	NO. OF SUB-LOTS	NO. OF INCREMENTS FROM EACH SUB-LOT
(1)	(2)	(3)
Up to 200 tonnes	2	20
201 „ 500 „	3	30
501 „ 2 000 „	4	40
2 001 tonnes and over	5	50

\*Methods for random sampling.

**D-3.3** Sampling from bulk shall be carried out as far as possible when the material is in motion. For this purpose the increments shall be drawn at regular intervals depending upon the rate of flow of the material of the sub-lot and the number of increments as given in col 3 of Table 4.

**D-3.4** All the increments from a sub-lot shall be pooled together to constitute a gross sample.

#### **D-4. SAMPLING FROM WAGONS**

**D-4.1 Lot** — In a single consignment all the material coming from the same source or supplier shall constitute a lot.

**D-4.2 Sub-lot** — For the purpose of sampling all the wagons in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal weight in accordance with Table 4.

**D-4.3** A minimum of 25 percent of wagons shall be selected at random from the sub-lot. The corresponding number of increments to be taken from a sub-lot shall be distributed over the selected wagons with a view to determining the number of increments that should be taken from each of the selected wagons in the sub-lot.

**D-4.4** All the increments taken from the selected wagons in sub-lot shall be pooled together to constitute a gross sample.

#### **D-5. PREPARATION OF SAMPLES**

**D-5.1** All the increments from the same sub-lot shall be thoroughly mixed to constitute a gross sample representing the sub-lot. The minimum size of the gross sample shall be about 3 kg. If the gross sample is less than this, additional number of increments shall be drawn so as to make up the required quantity.

**D-5.2** The gross sample shall be suitably reduced by the method of coning and quartering or any other suitable method to about 750 g which shall be divided into three portions called laboratory samples, one for the purchaser, another for the supplier and the third as the referee sample, each duly placed in the sample container.

#### **D-6. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY**

**D-6.1** One laboratory sample from each sub-lot shall be tested individually for all the requirements of this specification and the sub-lot shall be considered satisfactory if the laboratory sample satisfies all the requirements of this specification.

**D-6.2** The lot shall be declared to conform to the requirements of this specification if all the sub-lots are found satisfactory in **D-6.1**.

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